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EFFECT OF ANNEALING ON Cu-Nb-Sn SUPERCONDUCTING WIRE

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ABSTRACT

EFFECT OF ANNEALING ON Cu-Nb-Sn SUPERCONDUCTING WIRE. The most common application of superconductors is done in the form of superconducting wire. Among the existing types of superconductors, Cu-Nb-Sn superconductors are the most widely used as a wire, producing a high magnetic field. But the critical temperature (T_C) values of its superconductors are low enough so that the resulting magnetic field and its application fields are limited. In this study we investigated the effect of annealing treatment on the Cu-Nb-Sn superconducting wire. Note that the process of annealing on superconducting wire can increase the value of the critical temperature of 8K to 16K. The increase is predicted because of the forming of Nb_3Sn compounds, and the Nb_3Sn compound becomes more stable. Annealing processes were performed at temperatures ranging from 873K to 1173K as well as various annealing time from 32 hours to 120 hours. The superconductivity of the samples were analyzed using resistivity measurement by cryogenic system under low temperature condition. The annealing can be performed optimally at the temperature of 873K for 72 hours when T_C reaches 16K. However, the purity of the conductivity properties obtained at the optimal annealing temperature at 723K for 72 hours.

Keywords: Cu-Nb-Sn, Superconducting wire, Resistivity, Annealing, Residual Resistivity Ratio, Critical Temperature (T_C)

ABSTRAK

EFEK *ANNEALING* PADA KAWAT SUPERKONDUKTOR Cu-Nb-Sn. Aplikasi dari superkonduktor dilakukan dalam bentuk kawat superkonduktor. Di antara jenis superkonduktor yang ada, Cu-Nb-Sn adalah superkonduktor yang paling banyak digunakan sebagai kawat penghasil medan magnet tinggi. Tetapi nilai suhu kritis (T_C) superkonduktor yang cukup rendah menyebabkan medan magnet yang dihasilkan dan bidang aplikasi menjadi terbatas. Dalam penelitian ini menyelidiki efek perlakuan *annealing* pada kawat superkonduktor Cu-Nb-Sn. Dapat diketahui bahwa proses *annealing* pada kawat superkonduktor dapat meningkatkan nilai suhu kritis dari 8K ke 16K. Kenaikan ini diprediksi karena membentuk senyawa Nb_3Sn dan senyawa Nb_3Sn menjadi lebih stabil. Proses *annealing* dilakukan pada suhu berkisar antara 873K sampai 1173K serta berbagai waktu *annealing* dari 32 jam sampai 120 jam. Superkonduktivitas sampel dianalisis menggunakan pengukuran resistivitas listrik dengan memakai sistem kriogenik pada kondisi suhu rendah. *Annealing* dapat dilakukan secara optimal pada suhu 873K selama 72 jam saat T_C mencapai 16K. Namun, kemurnian sifat konduktivas listrik diperoleh pada suhu pendinginan optimal pada 723K selama 72 jam.

Kata kunci: Cu-Nb-Sn , Kawat superkonduktor, Resistivitas, *Annealing*, *Residual Resistivity Ratio*, Suhu kritis (T_C)

INTRODUCTION

Cu-Nb-Sn is the superconducting material that most widely used for applications as high magnetic field coils. This is due to the nature of this material that has the durability properties of superconductivity in a magnetic field, and also the nature of this material consisting of stable metal alloy. In the process of making this superconducting wire, Cu-Nb-Sn is basically done by using PIT (Powder-In-Tube) method that uses Cu as the sheath tube.

In a study of superconducting properties of Nb-Sn, the researchers usually have difficulty in resis-tivity measurements that require very low tempera-tures, which can only be done using a cryogenic equipment.

In the process of manufacturing supercon-ducting wire, annealing process is the final stage to get the higher and stable superconducting wire, and opti-mize the value of its TC [1]. This study was conducted to determine the optimal annealing process for Cu-Nb-Sn superconducting wire by resistivity mea-surement. Annealing process is known as an important step to improve the TC value, so that Nb3Sn compounds become stable.

EXPERIMENTAL METHOD

Preparation of the samples has been reported in our previous article [2]. In this research, we used variations in the annealing temperature of 723K, 873K, 1023K and 1173K [4-6,8]. We also made changes of annealing time to be 32, 72 and 120 hours.

The resistivity measurements were performed using four point probe method and Cryogenic System to create low temperature condition at Research Center for Metallurgy and Material, Indonesian Institute of Sciences.

Figure 1 shows photograph of the Cu-Nb-Sn superconducting wire we used. While Figure 2 shows the microscopic analysis at cross sectional of the wire. Cu layer is the outer layer that wraps around all monofilaments. Each monofilament made of Cu-Nb-Sn

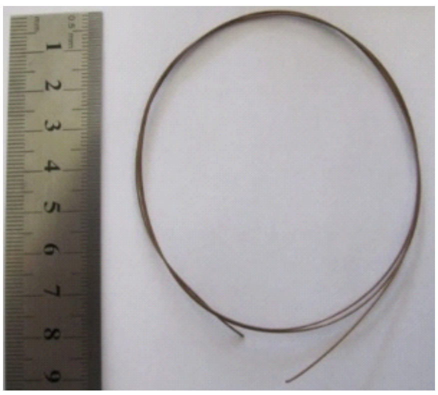

Figure 1. Photograph of Cu-Nb-Sn Superconducting wire (before annealing process).

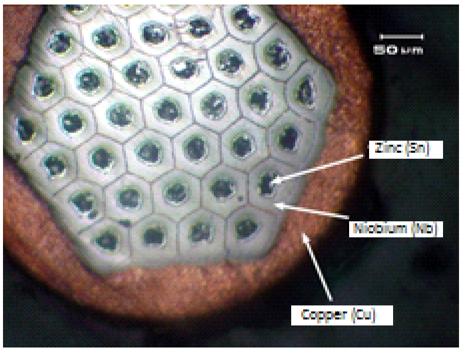

Figure 2. Sample of Cu-Nb-Sn Superconducting wire (before annealing process).

layers. Layer of Nb3Sn compounds were formed at boundary between Nb and Sn in each monofila-ment.[1,3,7,9]

RESULTS AND DISCUSSION

Figure 3 shows the results of the temperature dependence of resistivity measurements in the sample before the annealing process. In this graph, it appears that the value of T_C in superconducting wire is 8K. At temperatures above 50K, it shows the behavior of the conductor, and the temperature below 50K, it looks more

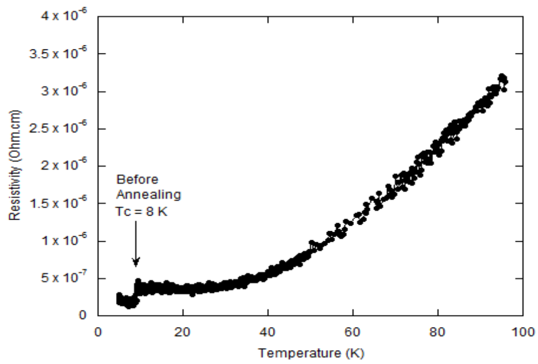

Figure 3. Temperature dependence of resistivity on Cu-Nb-Sn superconducting wires (before annealing process).

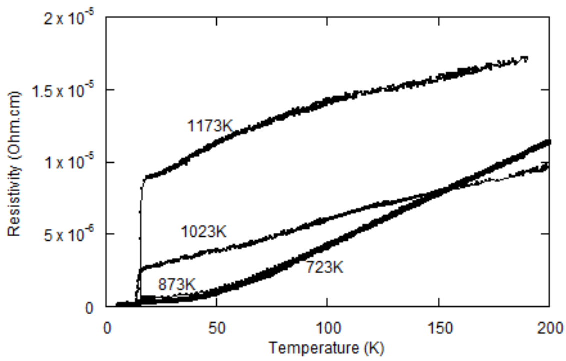

Figure 4. Temperature dependence of resistivity on Cu-Nb-Sn superconducting wires after annealing process (Annealing process were performed at 723K, 873K, 1023K and 1173K, for 72 hours).

flatten which indicates the existence of residual resistance of the sample.

The results of the annealing process with variation temperature are shown as Figure 4 and Figure 5. Annealing processes were done for 72 hours and at temperature of 723K, 873K, 1023K and 1173K. T_C for samples annealed at 723K, 873K, 1023K and 1173K were 14.5K, 16.5K, 15.5K and 16.0K, respectively (Figure 5(a)).

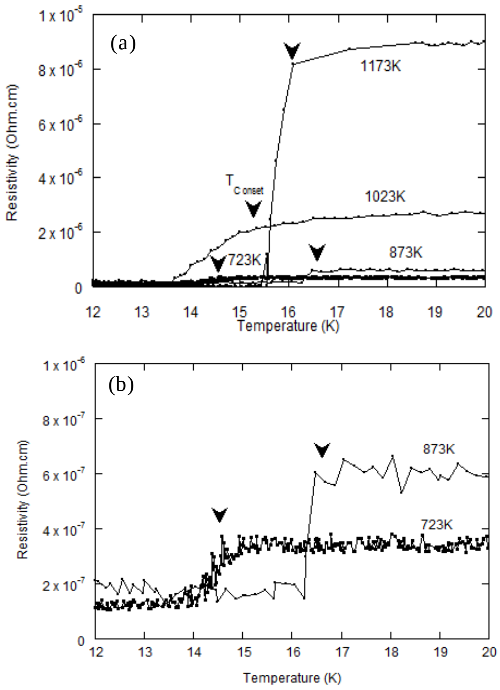

Figure 5. Temperature dependence of resistivity on Cu-Nb-Sn superconducting wires after annealing process (Annealing process were performed from 723K to 1173K, for 72 hours). The graph shows resistivity around TC. Figure 5.b shows the resistivity around TC at samples annealed at 723K and 873K.

Based on the results of the various annealing temperature, it is known that in order to obtain optimum TC, the annealing temperature should be 873K. At the annealing temperature of 723K, environmental conditions at the Nb_3Sn compound of superconducting wire is not stable yet, so that the TC value only reached 15.5K. However, at the annealing temperature of 1023K and 1173K, the TC value fell back from 16.5K into 15.5K and 16.0 K, respectively [1]. This is thought to occur because the annealing temperature is too high so that the Nb3Sn compound inside the superconducting wire become unstable [10,11]. Moreover, according to the resistivity above TC temperature, at samples heated with 1023K and 1173K, the curve of the resistivity is not linear. This is expected because of the diminishing of the conductivity. It is estimated that Cu layer becomes thinner and lead oxide reaction which cause the wires also have semiconductor properties.

The increase in annealing temperature to 1023K and 1173K does not lead to changes T_C values significantly, but causes the diminishing of Residual Resistivity Ratio (RRR) value of the samples (residual resistance of the samples becomes larger) [7,8]. It is estimated due to the oxides semiconductor that occurred as a result of the depletion of Cu layer.

In addition, this study also examined the changes of the resistivity at different annealing time. The annealing were done at 32, 72 and 120 hours. The results of the resistivity in the samples can be seen as Figure 6 .

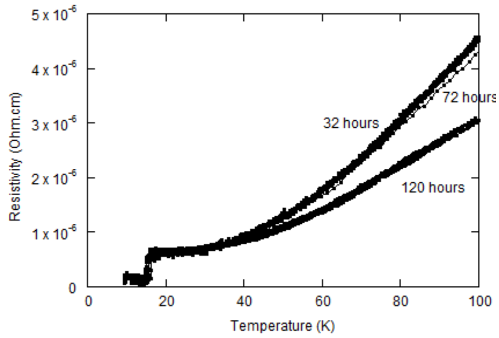

Figure 6. Temperature dependence of resistivity on Cu-Nb-Sn superconducting wires after annealing process (Annealing process were performed at 873K, for 32, 72 and 120 hours).

Figure 7 shows the temperature dependence of resistivity around T_C. Small arrows show the position of the T_C on each sample. T_C value for samples annealed for 32, 72, and 120 hours were 15.5K, 16.5K and 16.0K, respectively.

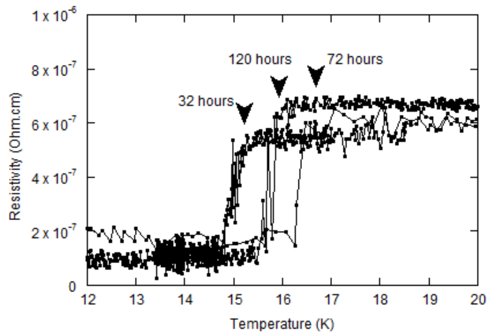

Figure 7. Temperature dependence of resistivity on around Tc

Based on the results of annealing with different annealing time, we obtained optimum conditions for annealing time was 72 hours. At the annealing time of 32 hours, the condition of the Nb_3Sn compound is not stable yet, the T_C value is also still at 15.5K, slightly different from the one heated for 72 hours. The resistivity at temperatures above T_C also showed no different behavior with the sample heated for 72 hours. This shows that the annealing time of 32 hours is still not optimal.

In sample annealed for 120 hours, T_C value achieves 16.0K, smaller than the sample annealed for

72 hours. In addition, the resistivity with temperature above T_C, it appears that the curve looks flatter compared to the sample annealed for 72 hours. It indicates a lower RRR value and the declining of conducting behavior. It is predicted due to long heating process so that impurities such as metal oxides were formed in the sample.

Table 1 shows a summary of the T_C value in the samples before and after annealing.

Table 1. T_C value of the samples

Annealing time	Annealing temperature			
	723K	873K	1023K	1173K
Before annealing	8.0 K			
32 hours		15.5 K		
72 hours	14.5 K	16.5 K	15.5 K	16.0 K
120 hours		16.0 K		

Table 1 shows the T_C value for each sample. From the results of various annealing temperature and time, it is known that the lack of annealing temperature causes the T_C does not reach the optimal value. While excessive annealing temperature does not affect the value of T_C significantly.

From the results of various annealing time, it is known that lack of annealing time also causes T_C does not reach the optimal value. While excessive annealing time also causes T_C does not reach the optimal value.

Table 2 shows the RRR value for each sample. RRR is calculated from the resistivity at room temperature (300K) divided by the resistivity before its T_C [7,8]. At the annealing temperature of 723K for 72 hours, the sample appeared to have the largest RRR value of 52.1, but when viewed its T_C value, annealing at 873K for 72 hours has the largest T_C of 16.5K. From this data, it can be seen that with the heating temperature of 723K, the sample has the best conductivity properties (annealing process improved the conductivity properties of the sample), but has not reached the optimal T_C value [4].

Tabel 2. RRR value of the samples

Annealing time	Annealing temperature			
	723K	873K	1023K	1173K
Before annealing	38.6			
32 hours		31.3		
72 hours	52.1	30.2	5.3	2.4
120 hours		19.0		

After heating at 873K, sample has an optimal T_C value, but the conductivity properties of the sample has been reduced compared to the sample annealed at 723K. In RRR data at various annealing time, it appears that the RRR value will decrease with the passing of the annealing process. However, the annealing process can increase the T_C value of samples.

Figures 8 shows the changes in the value of T_C and RRR at various annealing temperatures (annealing time remains for 72 hours). While Figure 9 shows the change in the value of T_C and RRR at various annealing time (annealing temperature remains at 873K). Figure 8 shows that T_C has a peak at a temperature of 873K, but RRR has a peak at a temperature of 723K. While in Figure 9 shows that T_C has a peak at 72 hours, RRR showed continuing decreasing in the length of annealing time.

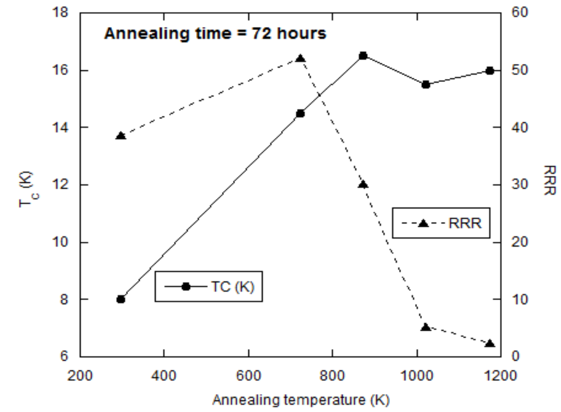

Figure 8. Plot of the changes in the value of T_C and RRR at various annealing temperatures (annealing time remains for 72 hours)

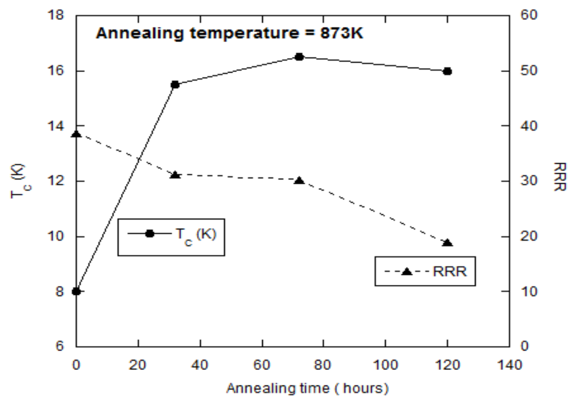

Figure 9. Plot of the changes in the value of TC and RRR at various annealing time (annealing temperature remains at 873K)

From the data of this study, it is noted that to obtain optimal T_C values, annealing at 873K for 72 hours is an appropriate parameter, but this also causes partially oxidized metal into the semiconductor. To obtain a superconducting wire with good conductivity properties, then annealed at 723K for 72 hour is the proper parameters [4].

Comparing Figure 8 and Figure 9, the changes of annealing time does not affect RRR value significantly, but the changes of annealing temperature affect RRR value significantly. This shows the settings on the annealing temperature of superconducting wires plays an important role to optimize the annealing process.

CONCLUSIONS

We have done annealing on Cu-Nb-Sn superconducting wire with different temperature and different annealing time. In this research, it is noted that the annealing can be performed optimally at the temperature of 873K for 72 hours. However, the purity of the conductivity properties obtained at the optimal annealing temperature at 723K for 72 hours. Lack of annealing temperature causes the T_C value does not reach the optimal value. While high temperature annealing causes the conductivity properties of superconducting wire which is much reduced (RRR value is decreased).

Lack of annealing time also causes the T_C does not reach the optimal value. Excessive annealing time also causes declining in the T_C value and leads to reducing of the metal purity (reducing of RRR value). Annealing temperature plays an important role in the optimization of the annealing process. So, setting the right temperature is a necessary condition in the annealing process of Cu-Nb-Sn superconducting wire.

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